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(54) Title: ICE CONFECTIONS CONTAINING CLEAVED KAPPA-CASEIN

(57) Abstract

The properties of ice cream confections containing milk proteins are improved by cleaving the Kappa-casein protein component with a milk clotting enzyme.

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ICE CONFECTIONS CONTAINING CLEAVED KAPPA-CASEIN

FIELD OF THE INVENTION

This invention relates to ice confections, particularly aerated compositions, containing milk protein. There is no restriction on the route by which this protein can be added to the confections, thus it may be incorporated as liquid milk, cream, skimmed milk, milk powder and skimmed milk powder, as examples. The invention is particularly applicable to ice cream, but is also usable with ice milk, frozen yoghurts and frozen custards. These confections are prepared at low temperatures and intended for consumption while in the frozen state.

BACKGROUND TO THE INVENTION

The composition of ice confections have developed over the years to include a number of ingredients to provide specific desirable product properties. However, in recent times there has been a general desire by consumers to have products with a natural base, that is incorporating only the basic traditional ingredients. For ice cream these traditional ingredients would be cream, milk, sugar and, optionally, flavour.

The manufacturer of ice confections now has the problem of preparing a product matching recent products containing functional ingredients, but using a restricted list of ingredients. Ice confections, as developed in recent years, can be expected to contain such functional ingredients as gelling agents, eg gelatin and locust bean gum (LBG), emulsifiers and emulsion stabilisers.

LITERATURE

Ice confections have been well characterised in the literature and general disclosures will be found in Arbuckle and J Soc Dairy Technology 1990, 43(1), pp 17-20.

US 4 626 441 (Wolkstein) describes the use of aspartame and other sweeteners and bulking agents in frozen desserts. Rennet is, together with a number of other materials, quoted as a bulking agent. There is no identification of a milk clotting enzyme with the benefit of emulsifier and stabiliser properties. The criticality of the degree of cleavage is not disclosed.

GENERAL DESCRIPTION OF THE INVENTION

The invention is particularly directed to the replacement of the stabiliser and emulsifier components. This is achieved by the presence of an effective amount of a milk clotting enzyme capable of cleaving the kappa-casein protein present in the milk. The applicants have found that cleaving from about 15% to about 70% of the casein, preferably from about 20% to about 60%, generates a species providing the emulsifier and stabiliser functions. Below 15% cleavage the benefit of the invention is not found while above 70% the cleavage will lead to coagulation of the protein. The presence of stabilisers in ice cream will provide two physical properties, ie viscosity of the premix and the meltdown stability. The addition of a milk clotting enzyme provides a premix viscosity comparable to that obtained with a standard stabiliser. The presence of the enzyme also gives improved meltdown properties, with increased resistance to melting. Thus use of the invention provides an ice confection with stability of structure during consumption.

The degree of cleaving required in the kappa casein ingredient may be obtained by mixing two sources of kappa-casein. Usually a single source of K-casein will be

cleaved to the desired degree, but combining an uncleeaved source with a source cleaved to more than 70% is also effective. This latter source could be 100% cleaved and would be unsatisfactory if used as the sole source. When mixed with a source of K-casein which is uncleaved, or cleaved to a low degree, this highly cleaved source would function to be effectively a K-casein having cleavage in the range 20% to 70%. In some production systems use of a mixture may be easier to use and monitor.

The structure of the ice cream or other ice confection is also altered by having the cleaved Kappa casein present; this is demonstrated by the size of the fat droplets which can be retained in stable emulsion.

The ice confections containing cleaved kappa-casein have been found to have acceptable consumer properties. The enzymic cleaving by the milk clotting enzyme can be monitored by measuring the release of the peptide GMP. The class of milk clotting enzymes is well characterised and includes rennet, microbial enzymes and those obtained by genetic engineering.

The kappa-casein cleaves to give para-kappa-casein and macropeptides. The enzyme altered milk protein gels in the presence of the calcium ions naturally present.

Modilase S (obtainable from Chr Hansen's Laboratorium A/S) is an example of an effective enzyme.

TEST METHODS

The following methods were used in the testing and evaluation of the products according to the invention.

i) Casein cleaving - the degree of cleaving can be monitored by measuring the release of glycomacropeptide (GMP) during the enzyme reaction. This release is followed quantitatively using gel permeation chromatography (GPC)

high performance columns.

A Varian 9050 variable UV VIS detector is used in combination with a Varian 910 Solvent Delivery System, using a TSK-Gel G2000SW_{XL} 7.8mm x 30cm column and a TSK SW_{XL} columnguard 7.8mm x 4cm.

The eluent consists of potassium hydrogen phosphate (1.74g), potassium dihydrogen phosphate (12.37g) and sodium sulphate (2.41g) dissolved in 1000ml of double distilled water.

1ml of sample is manually injected into a Rheodyne valve fitted with a 20 μl loop. The flow rate was set at 1.0ml/minute, and the detector set at 214nm.

The GMP peaks are analysed using a Munícham analysis (version 1.65) package, which calculates these areas taking into account any change in the baseline at the beginning and end of the chromatographic run.

Preparation of the enzyme/SMP solution for Gel Permeation Chromatography: The SMP and enzyme solution is incubated at set temperature and time. The enzyme is then inhibited by the addition of trichloroacetic acid. The mixture is filtered using Whatman hardened 54 paper. Prior to injecting the sample into the machine it is passed through a second filter with a pore size of 0.2μm. The method used in this study is explained in detail in Hooydonk ("The renneting of milk" Thesis, Agricultural University, Wageningen, Netherlands) and involves the use of 8% trichloroacetic acid to precipitate selectively the whey proteins. Whey proteins have similar retention times to GMP but unlike GMP they absorb UV light at a wavelength of 280nm. By monitoring the chromatogram at a wavelength of 280nm any potential interference from whey protein can be determined.

ii) Viscosity - the apparent viscosity of the premix was

measured at 5°C using a Carrimed viscometer at $100s^{-1}$ as Pascal seconds (Pas).

iii) Meltdown of the ice cream product was measured by the procedure of exposing the product, at a temperature of -20°C, to ambient, ie 25°C, on a metal mesh. The initiation time (in minutes) is the time for the first 4% of the weight loss to occur; the mass loss(%) is the mass lost after 4 hours as a %age of the original mass. The drip rate (wt%/hr) is the average rate at which the weight of liquid collected increases after the initiation time has elapsed, up to a total of 4 hours.

iv) Fat droplet - the fat droplet size in the premix was measured by a laser light scattering device (Malvern Mastersizer) and is quoted as the volume moment mean diameter D[4,3] in microns.

SPECIFIC DESCRIPTION OF THE INVENTION

Examples will now be given to illustrate but not limit the invention.

A basic ice cream formulation with the following composition was prepared.

Ingredient	weight(%)
SMP	9.00
sucrose	16.00
cream(48% fat)	26.00
flavour	0.27
water	48.73

A solution of SMP (4.5kg) in water (24.365kg) was heated to the set temperature of 37°C and an enzyme solution of Modilase S14 (25mls) in 1000mls water added with stirring.

That is, 10g enzyme solution is used per Kg of SMP solution. The mixture was incubated at 37°C for 5 minutes and then heated rapidly to 83°C and retained at that temperature for 15 minutes to deactivate the enzyme. From standardisation experiments it was known this procedure gave a kappa-casein cleavage of 50%. The sucrose (8kg) and cream (13kg) were then added and homogenised into the mixture. This forms the ice cream premix. The mixture was then pasteurised, cooled, aged and frozen, with the product being aerated to an overrun of 110% during freezing.

Example I

The apparent viscosities of ice cream premixes containing enzyme or locust bean gum (LBG) were measured and are given in Table I.

Table I

Ingredient	visc (up) Pa s	visc (down) Pa s
Modilase	0.65	0.25
LBG (0.1%)	0.07	0.07
LBG (0.5%)	0.78	0.77
None	0.03	0.03
Commercial target	0.50	0.40

It is seen the use of a milk-clotting enzyme provides viscosities comparable with that of commercial ice creams.

The meltdown characteristics of ice cream products were measured and are given in Table II.

Table II

Ingredient	Initiation (mins)	Drip rate (%/hr)	Mass loss (%)
Modilase	90	20.0	51.0
LBG (0.1%)	30	26.0	77.0

LBG (0.5%)	77	10.5	33.2
None	22	25.4	86.4

It can be seen the use of milk-clotting enzyme provides melt down which approximates to that obtained with LBG. The initiation time has improved over the sample which contains no additive.

The fat droplet size of premixes containing enzyme or a standard emulsifier Admul 4103 (obtainable from Quest Ltd of Ashford, England) were measured and are shown in Table III.

Table III

Ingredient	D[4,3] micron
Modilase	15.0
Admul (0.1%)	0.8
Admul (0.5%)	0.8
None	1.0
Commercial	1.1

Thus the use of a milk-clotting enzyme provides larger fat droplets in a stable emulsion.

Example II

Example I was repeated but using kappa casein cleaved to one of several levels. The melt down properties of the resulting ice creams are shown in Figure 1. The kappa casein feedstocks are represented in Figure 1 as follows:

Curve	Degree of cleavage
I	zero (control)
II	25
III	50
IV	75
V	100

Figure 1 shows the weight loss (as a percentage) plotted against time; it is seen the use of kappa-casein cleaved in the range from about 20% to about 70% provides the preferred range of melt down properties.

CLAIMS

1. A method of preparing an ice confection containing kappa-casein as a component of milk protein wherein the kappa-casein is contacted with a milk-clotting enzyme at a temperature and for a time sufficient to cleave from about 15% to about 70% of the kappa-casein in the protein.

2. A method according to claim 1 wherein the kappa-casein is cleaved from about 20%.

3. A method according to claim 1 or 2 wherein the kappa casein is cleaved to about 70%.

4. An ice confection containing kappa-casein cleaved from about 15% to about 70% by a milk-clotting enzyme.

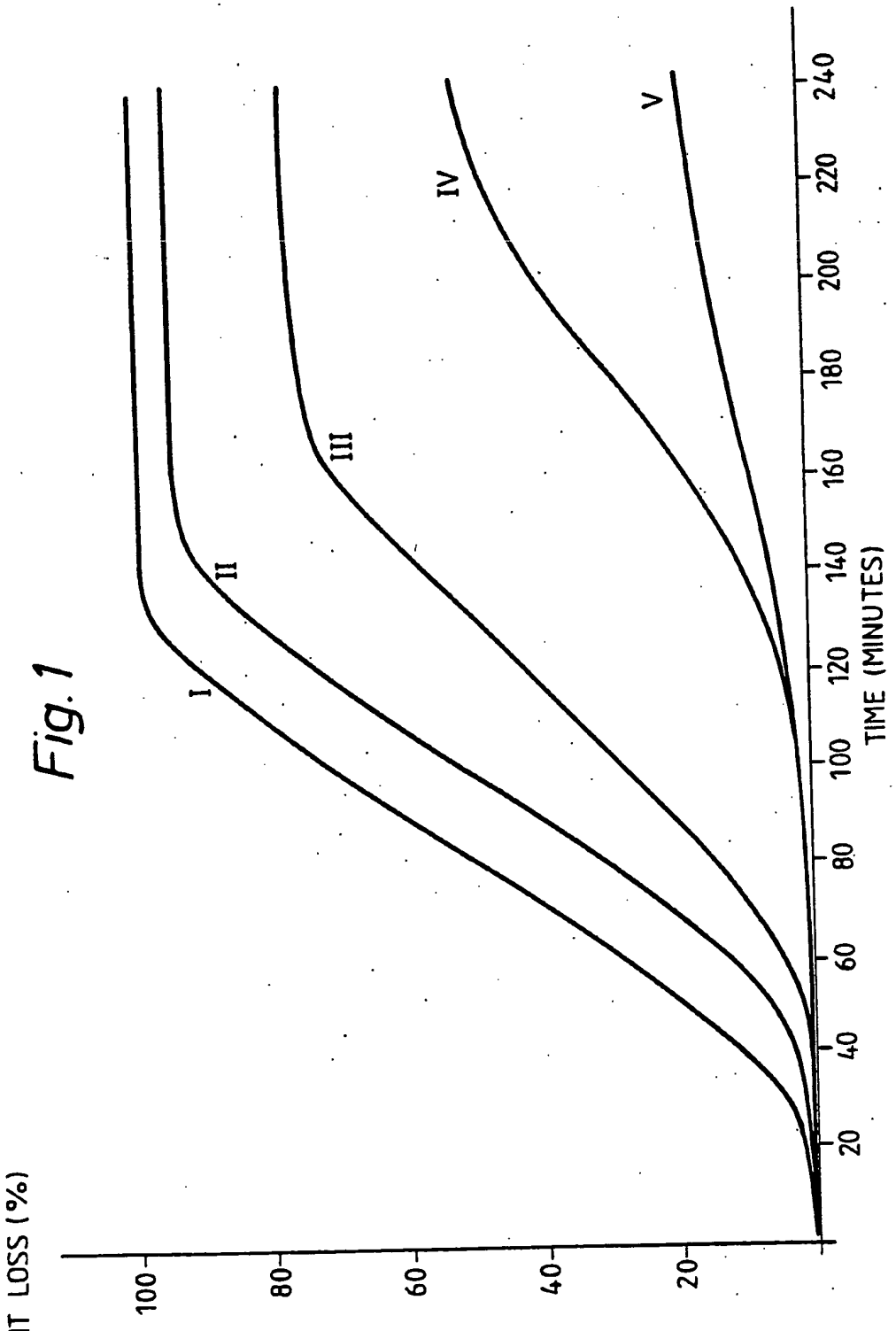
WEIGHT LOSS (%)
100
80
60
40
20
TIME (MINUTES)
20
40
60
80
100
120
140
160
180
200
220
240
I
II
III
IV
V

Fig. 1

INTERNATIONAL SEARCH REPORT

International Application No PCT/GB 93/02242

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int.Cl.5 A 23 G 9/02

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
Int.Cl.5	A 23 G A 23 J

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched[8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category °	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No.[13]
X	FR,A,2100161 (LENDERINK & CO. N.V.) 17 March 1972, claims 1-3,6 ---	1-3
X	US,A,3889001 (N.F. BUIDE et al.) 10 June 1975, claims 6,8 ---	1
A	GB,A,2021921 (UNILEVER LIMITED) 12 December 1979, page 1, lines 40-51,66-76 ---	1-3
A	JOURNAL OF AGRICULTURAL & FOOD CHEMISTRY, vol. 36, no. 5, September 1988, pages 883-892, Washington, DC, US, J.-M. CHOBERT et al.: "Solubility and emulsifying properties of caseins ...", pages 887-891 --- -/-	

° Special categories of cited documents : [10]

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

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IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
22-02-1994	16. 03. 94
International Searching Authority	**Signature of Authorized Officer**
EUROPEAN PATENT OFFICE	Ingrid Dagmar FRANK

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III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category °	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	FOOD SCIENCE AND TECHNOLOGY ABSTRACTS, AN = 85:7268, DN = 85-05-P0031, International Food Information Service, Berkshire, Reading, GB, & JOURNAL OF THE SOCIETY OF DAIRY TECHNOLOGY, vol. 37, no. 4, 1984, pages 119-121, J. Rothwell: "Uses for dairy ingredients in ice cream ...", abstract ---	
A	FOOD SCIENCE AND TECHNOLOGY ABSTRACTS, AN = 81:14050, DN = 81-10-P1779, International Food Information Service, Berkshire, Reading, GB, & ZUIVELZICHT, vol. 73, no. 8, 1981, pages 156-158, F.M.W. VISSER: "Uses of milk protein in the food industry", abstract -----	

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO. GB 9302242

SA 81170

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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		DE-A- 2132444	05-01-72
		GB-A- 1313807	18-04-73
		NL-A- 7009789	04-01-72
US-A- 3889001	10-06-75	None	
GB-A- 2021921	12-12-79	AT-B- 362986	25-06-81
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		US-A- 4259357	31-03-81

CORRECTED VERSION*

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